

Characterization of Volatiles Loss from Soil Samples at Lunar Environments

Julie Kleinhenz

NASA Glenn Research Center, Cleveland, Ohio, 44135

Jim Smith

NASA Kennedy Space Center, Florida 32899

Ted Roush, Anthony Colaprete

NASA Ames Research Center, Mountain View, CA 94035

Kris Zacny, Gale Paulsen, Alex Wang

Honeybee Robotics Spacecraft Mechanisms Corporation, Pasadena, Calf. 91103

Aaron Paz

NASA Johnson Space Center, Houston, TX, 7/958

Resource Prospector



Mission

- Prospect for water at the lunar poles as a potential resource for In-Situ Resource Utilization (ISRU)
- Characterize the nature and distribution of water/volatiles in the lunar polar sub-surface materials



Mobility

Rover

- · Mobility system
- Cameras
- Surface interaction

Sampling

Drill

- Subsurface sample acquisition
- Auger for fast subsurface assay
- Sample transfer for detailed subsurface assay

Processing & Analysis

Oxygen & Volatile Extraction Node (OVEN)

- Volatile Content/Oxygen Extraction by warming
- Total sample mass

Lunar Advanced Volatile Analysis (LAVA)

- Analytical volatile identification and quantification in delivered sample with GC/MS
- Measure water content of regolith at 0.5% (weight) or greater
- Characterize volatiles of interest below 70 AMU

Prospecting

Neutron Spectrometer System (NSS)

 Water-equivalent hydrogen > 0.5 wt% down to 1 meter depth

NIR Volatiles Spectrometer System (NIRVSS)

- Surface H2O/OH identification
- Near-subsurface sample characterization
- Drill site imaging
- Drill site temperatures

Resource Prospector (RP) Integrated Thermal Vacuum Test Program



- A series of ground based dirty thermal vacuum tests are being conducted to better understand the subsurface sampling operations for RP
 - Volatiles loss during sampling operations
 - Hardware performance
 - Concept of operations
- 5 test campaigns over 5 years have been conducted with RP hardware with advancing hardware designs and additional RP subsystems
 - Volatiles sampling 4 yrs
- Using flight-forward regolith sampling hardware, empirically determine volatile retention at lunar-relevant conditions
 - Use data to improve theoretical predictions
 - Determine driving variables for retention
 - Bound water loss potential to define measurement uncertainties





- The main goal of this talk is to introduce you to our approach to characterizing volatiles loss for RP.
 - Introduce the facility and its capabilities
 - Overview of the RP hardware used in integrated testing (most recent iteration)
 - Summarize the test variables used thus far
 - Review a sample of the results



VF13 Planetary Surface Simulation Facility



Dedicated 'dirty' thermal vacuum chamber operated with up to 1-ton of lunar soil simulant

Dimensions

- Maximum internal volume of 6.35 m³
- Internal dimensions: 3.6 m tall, 1.35 m diameter with cold wall, 1.5 m without cold wall
- Fixed base 1.08 m deep + Removable cap 2.52 m tall

 Thormal capability

Thermal capability

- Removable cold wall in cap (top 2.5 m of chamber)
 - Temperature control from ambient to liquid nitrogen temperatures
 - 2 semi circular halves, independently controlled to achieve temperature gradients
 - Minimum temperature 80K (liquid nitrogen cooled)
- Fixed base has separate Liquid Nitrogen cooling, independent of cold wall
 - Supports cooling of soil bin (existing bin is 0.278 m diameter, 1.2 m tall)
- Liquid nitrogen is supplied from a 55,000 gallon dewar

Vacuum capability

- Achievable pressure on the order of 10⁻⁶ Torr, with soil
- Variety of customizable electrical and mechanical feed-throughs
- Four vacuum pumps to accommodate range of pressure regimes and pump rates
- (in process) Mars gas capability: Flow panel controlled with a Mass Spectrometer to maintain a Mars environmental conditions.

Facility operation

- PLC control allows for unattended operation for majority of pump down and cooling
- Customizable digital data acquisition system supporting over 80 channels
- Internal cameras for optical access







VF13 Research Hardware



Cylindrical Bin ("Drill Tube")

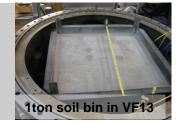
- 1.2m (48in) tall, 0.278m (11in) diameter
- Holds 100 kg of simulant
- Three side ports for soil embedded thermocouples (15, type T)
- Clamp on LN₂ Coolant system, soil temperature as low as -160° C





Square Bin

- 1 m x 1 m x 1 m
- Holds 800 kg of soil simulant



Robotic Translation Table (trolley)

- Enables lateral motion of research hardware to reach different locations on the soil bed surface
- Individual, Manual control of X and Y directions
- Position Encoders : ± 2 mm (approx.)



Soil Bin analysis methods



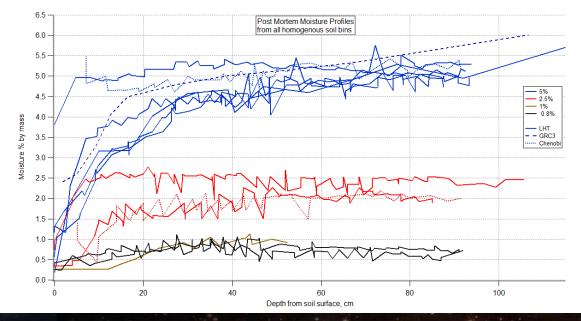
Soil Bin Preparation

- LHT-3M, ~100kg, doped with distilled water
 - Mixed in batches of 20- 25kg (5 gal)
 - Samples taken from each batch to verify moisture
- Compacted into cylindrical bin
 - Vibratory compaction with 150lb surcharge weight
 - Compacted in layers, ~20 kg each

"Post Mortem"

- No in-situ moisture measurement during vacuum test
- Depth dependent moisture profile generated after test (thawed soil bin) using core sampling
 - Difference between thawed and frozen bin moisture profile only impacts the top ~10cm
- Majority of desiccation occurs in top 30cm

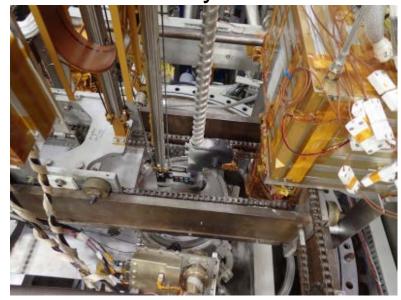




RP Test hardware



- ★RP EDU Drill: Honeybee Robotics
- ★Near InfraRed Volatiles Spectrometer System (NIRVSS): NASA ARC
- **★**Oxygen & Volatile Extraction Node (OVEN): *NASA JSC*
- Sample Capture Mechanisms
- Residual Gas Analyzer





Sample analysis methods





Test samples

- Sampling at 30 to 40cm depth
- Drilling in progressive 10cm bites
- The bottom 10 cm of auger captures sample on tapered auger flutes
- Sample dispensed into crucibles using a passive brush wheel and funnel on drill
- Solenoid actuated, spring driven seal mechanism with a knife edge-to-teflon seal, 100lbf clamp force

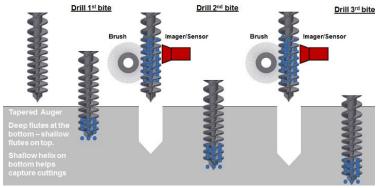
Sample analysis

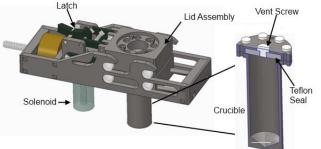
Moisture content of each sample is measured

using ASTM standard

- Bake at 110° C
- Weight change









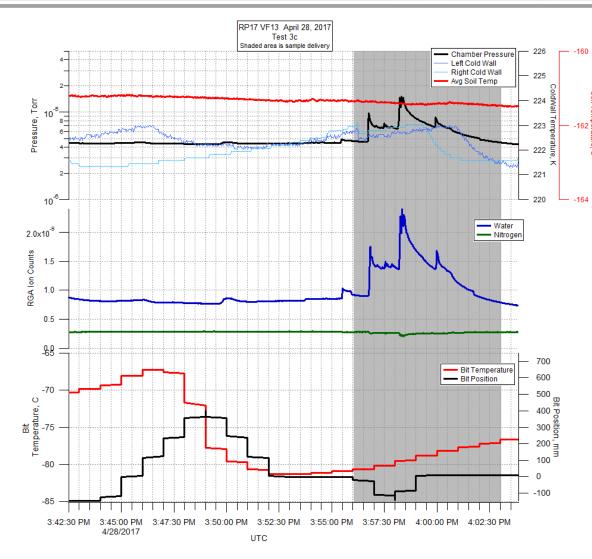
Variables



Pressure	Low as possible	1e ⁻⁵ Torr to 2e ⁻⁶ Torr
Shroud Temperature	Controlled	-50° C to -175° C
Soil Bin Temperature	Low as possible (Dependent on time, soil moisture)	-80° C to -163° C
Soil Bin Moisture	Controlled	≤ 5wt%
		Stratification
Sample Crucible Temperature	Controlled	10° C
	Cold as possible	-20° C to -70° C
Sample exposure time (in crucible)	Controlled	3min delay
	Fast as possible	~5min
Sample size	Target 15 g	Average 12g (Range 4 g to 20 g)

Test Results, example





Results from a drill sample bite:

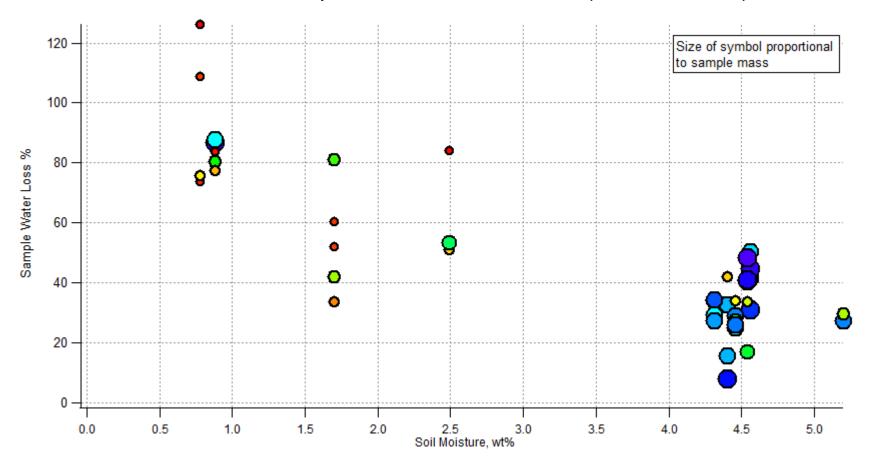
Water release, according to RGA, show majority of release occurs when dispensing into the sample crucible.

Sample 2017_O3: 3.3g, 0.4wt%, 84% loss

Test Results, general observations



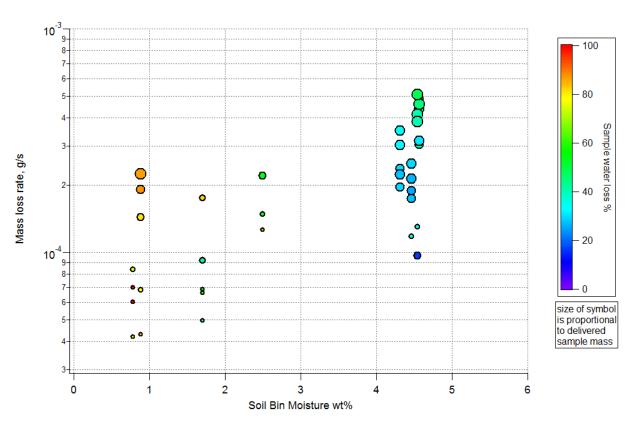
- Samples from a higher soil content retain higher percentage of water
- Data from low mass samples is less consistent (more scatter)



Test Results, general observations



- The rate of mass loss appears to be consistent for similar sample sizes. Samples with higher starting moisture content therefore loose less %.
- This mass loss could be correlated to sublimation rate.
- The sample is exposed to 4 temperatures: which is the driving temperature?
 - Soil bin
 - Cold wall
 - Drill bit
 - Sample Crucible



Test Results, general observations



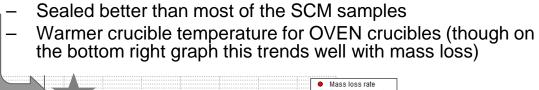
The closest correlations are with:

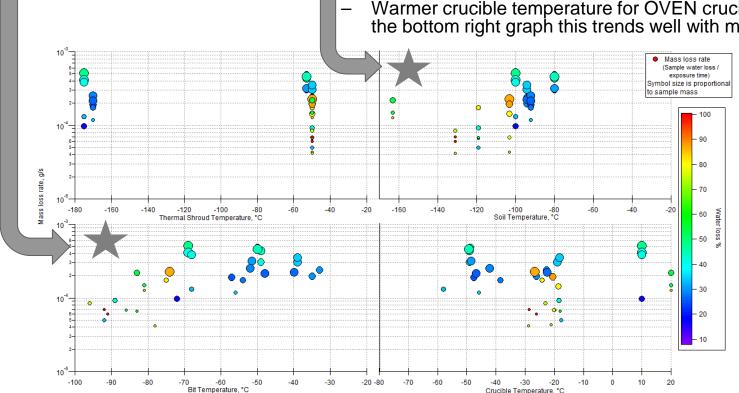
Bit temperature

But the lower temperature also have lower sample masses.

Soil temperature:

The 3 points at the lowest temperature are outliers to this trend. These are the 3 OVEN samples whose differences are:





Summary



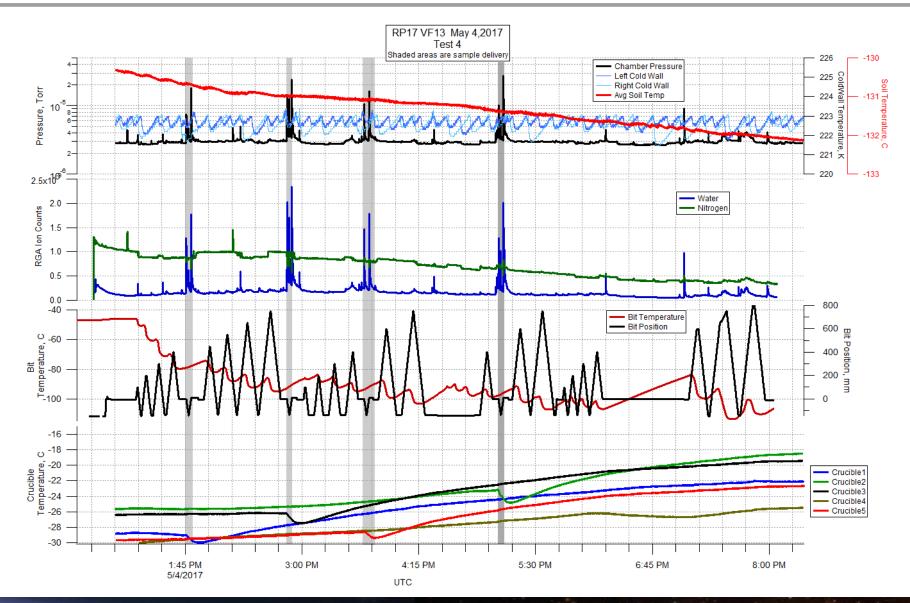
- To date we have conducted 4 test campaigns with volatiles sampling
 - 43 samples total
 - 4 soil moisture conditions with 26 samples from the same (~5wt%)
- Test performed with 3 RP subsystems: Drill, NIRVSS, OVEN
 - 3 tests were performed with the RP OVEN hardware, all the rest with the customized Sample Crucible Mechanisms (SCMs)
- Using flight-forward regolith sampling hardware, empirically determine volatile retention at lunar-relevant conditions
 - Use data to improve theoretical predictions
 - Determine driving variables for retention, adjust hardware and con-ops accordingly
 - Bound water loss potential to define measurement uncertainties
- Analysis of sample results in on-going, with a summary paper expected at the ASCE Earth and Space conference, April 2018



BACKUP

Test Results, example





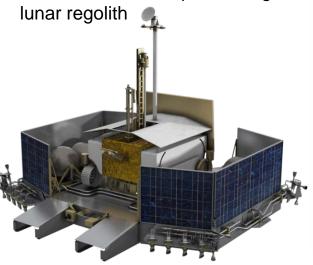
Resource Prospector (RP) Overview



Mission:

Characterize the nature and distribution of water/volatiles in lunar polar sub-surface materials

Demonstrate ISRU processing of





RP Specs:

Mission Life: 6-14 earth days (extended missions being studied) Rover + Payload Mass: 300 kg

✓ FY15: Phase A (Demonstration: RP15) Total system wet mass (on LV): 5000 kg Rover Dimensions: 1.4m x 1.4m x 2m

Rover Power (nom): 300W

Customer: HEOMD/AES Cost: ~\$250M (excl LV)

Mission Class: D-Cat3

Launch Vehicle: EM-2 or ELV

Project Timeline:

- ✓ FY13: Pre-Phase A: MCR (Pre-Formulation)
- ✓ FY14: Phase A (Formulation)
- ✓ FY16: Phase A (Risk Reduction)
- FY17: L2 Requirement Lockdown (July 11)
- FY18: MRD and PDR (Implementation)
- FY19: CDR (Critical design)
- FY20: I&T
- FY21: RP launch

